

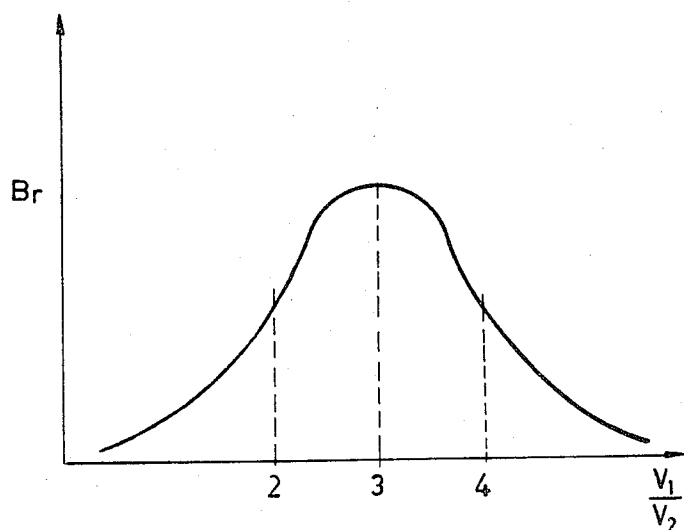
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METHOD OF CHEMICALLY POLISHING IRON, ZINC AND ALLOYS THEREOF

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METHOD OF CHEMICALLY POLISHING IRON, ZINC AND ALLOYS THEREOF

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4 Claims. (Cl. 106—3)

ABSTRACT OF THE DISCLOSURE

Polish iron, zinc and alloys of these metals by use of aqueous solution of hydrogen fluoride and hydrogen peroxide. This abstract is not intended to be a description of the invention defined by the claims.

The surface of a metal on which a metal coating is to be deposited by reduction, exchange or electro deposition, must have a suitable surface condition in order that the deposition is effected under favourable conditions.

In general, the shining appearance of the metal surface is an indication that the surface condition is particularly suitable for providing such deposits. When this condition is met, the adherence of the deposited metal is improved and the metal surface obtained by the deposition, which itself is shining, presents a great resistance against corrosion. The solderability of such thin films which are deposited on a shining substrate is better than that of the films which are deposited on a dull metal.

In addition the shining appearance of a metal surface is a criterion for the resistance against corrosion and may be used to obtain decorative effects.

A method of electrolytically polishing zinc by means of acid or basic solutions in an oxidizing medium is known as well as that for obtaining a polished surface of titanium, zirconium and alloys thereof by means of a mixture containing hydrogen fluoride.

When treating the surface of articles which partly consist of different alloys, the chemical polishing may be difficult if not impossible. The various metals and alloys do not behave in the same manner with respect to the same acid solution. If, in addition, the various alloys are welded to one assembly, this remains electrochemically heterogeneous and the difference in tension at the contact places between the various components as a rule involves a preferred chemical attack.

The method of chemically polishing according to the invention is particularly suitable for homogeneous components of iron, nickel or cobalt and also in the case that components of the same metals are combined heterogeneously. For applying the invention to the polishing of other alloys it is sufficient to vary the ratios of the composing parts of the polishing bath by adding, if desired, additives suitable for the purpose in view. The invention may be applied in particular to carbon steels and may be used for zinc.

According to the invention, the method of polishing the surface of metals or oxidizable metal alloys, and in particular zinc or analogous metals, is characterized in that the surface to be polished is immersed for a restricted period of time in an aqueous solution of hydrogen fluoride and hydrogen peroxide.

According as the metals or alloys are more or less oxidizable, the treatment of the metal surfaces takes place in the cold or in the heat, the optimum temperature for polishing being higher for metals which are less oxidizable and being determined for each individual case. Also the concentration of hydrogen peroxide is proportionally

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larger according as the metal or the alloy to be polished is less oxidizable.

For the rest, the concentration of hydrogen fluoride may be rather weak so that the polishing operation with solution according to the invention can take place in a glass container or in a container which comprises a glass window through which the polishing process can be followed

Generally the ratio of molecular concentration of hydrogen peroxide-hydrogen fluoride lies between 3 and 7.

The figure in the drawing shows how the shine of the surface of an alloy varies as a function of the ratio of the concentration of hydrogen peroxide and of hydrogen fluoride.

Preferably, the starting products of the solution are concentrated and given the dilution which best corresponds to the metal to be polished. These products may consist of a solution of 48 to 50% by weight of hydrogen fluoride, i.e. containing approximately 25 g. mol. of hydrogen fluoride per litre, and a solution of hydrogen peroxide of 30%.

Preferably, for example, zinc, iron and carbon steels are treated while cold. On the contrary, iron, nickel, cobalt are polished at a temperature of the order of 80° C. Iron-nickel-cobalt alloys and iron-nickel alloys are polished at a temperature of approximately 70° C.

When the activities are carried out in the heat, the components to be polished are transferred to the container with the solution according to the invention only when the temperature of the latter has been reached and heating has been stopped.

Also the period of treatment itself must be determined for each individual case but it often is in the order of 1 minute.

Example

First a solution of hydrogen fluoride is prepared by diluting 210 ml. of the starting solution of hydrogen fluoride 48 to 50% to 1 litre by deionized water. 300 ml. of this solution are added to 1 litre of the starting solution of hydrogen peroxide of 30%.

The resulting mixture is then diluted with deionized water, for example with 1300 ml., and heated to a temperature of approximately 80° C. in the polishing container which preferably consists of glass. Then heating is discontinued and the components to be polished, for example, components of iron, nickel, cobalt or an alloy of these metals, is immersed in this bath for 1 minute.

The quality of the resulting polish depends upon the ratio of the concentrations of hydrogen peroxide and hydrogen fluoride and it has been found, that for polishing iron, nickel, cobalt and alloys of iron-nickel a maximum shine is obtained for a molecular ratio of hydrogen peroxide to hydrogen fluoride which is approximately 5 which is the case noticeably with the said solution in the example.

As shown in the figure the shine or reflection power of the polished surface varies indeed as a function of the ratio of the concentrations of hydrogen peroxide and hydrogen fluoride. In the case of the alloy iron-nickel-cobalt the maximum shine is obtained for a volume ratio V_1/V_2 of the above components which equals 3 which corresponds to a molecular ratio of 5. The polishing operation is preferably carried out with solutions in which the volume ratio of hydrogen peroxide to hydrogen fluoride lies between 2 and 4 which corresponds to values 3.1 and 6.3 of the molecular ratio.

The action of the solution on the component to be polished is stronger on the projecting points of the surface where the catalytic decomposition of the hydrogen peroxide is greater. This increased oxidation also involves an increased action of the acid.

With the use of this method shining surfaces can easily be obtained even in the case of mixed components, i.e.

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component parts which are composed of elements of different alloys.

In addition, in the composition of the solutions used within the scope of the invention of which the ratio of the components may vary the concentration of hydrogen fluoride is only approximately 1% so that this involves no risks in operation.

As already noted above, articles consisting of iron or iron alloys, for example, iron-nickel-cobalt or iron-nickel, must be treated at elevated temperature. Polishing certain metal component parts for electronic devices sometimes involves difficulties. Some component parts are melted to glass component parts which, when treated with a warm mixture of hydrogen fluoride with hydrogen peroxide, are attacked. The gas-tight and vacuum-tight properties of such a seal which are of great importance for an electronic device, may be adversely influenced by it.

According to a further elaboration of the method according to the invention it is possible to subject iron alloys, for example, iron-nickel and iron-nickel-cobalt, to a polishing treatment at room temperature, namely by a suitable pretreatment of the surface to be polished before it is immersed in the aqueous mixture of hydrogen peroxide and hydrogen fluoride.

This pretreatment consists in that the articles are kept immersed for a restricted period of time in a mixture of chlorides and tartrates, at an elevated temperature which lies below the boiling point of the solution.

An example of such a pretreatment bath consists of a solution which contains per litre: 300 g. of tartaric acid and 6 mol of hydrochloric acid. This bath is preferably heated at a temperature of approximately 60° C. The time of treatment is approximately 6 to 8 minutes.

What is claimed is:

1. A method of polishing the surface of a metal object selected from the group consisting of iron, zinc and ox-

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idizable alloys thereof said method comprising immersing said metal object for a short period of time in an aqueous solution of hydrogen peroxide and hydrogen fluoride, the molecular ratio of hydrogen peroxide to hydrogen fluoride being between about 3:1 and 7:1.

2. The method of claim 1 wherein the metal objects are oxidizable iron alloys selected from the group consisting of nickel-iron and nickel-iron-cobalt alloys.

3. The method of claim 2 wherein the metal objects are pretreated prior to the treatment with the aqueous mixture of hydrogen peroxide and hydrogen fluoride by being heated in an aqueous solution of chlorides and tartrates.

4. The method of claim 1 wherein the solution of hydrogen peroxide and hydrogen fluoride is formed by diluting with water a solution of 50% by weight of hydrogen fluoride and a solution of 30% by weight of hydrogen peroxide.

References Cited

UNITED STATES PATENTS

3,103,733	9/1963	Fauro et al.	204—143
3,158,517	11/1964	Schwarzenberger ...	252—79.3
3,228,816	1/1966	Kendall	204—140.5
2,689,785	9/1954	Simon	156—20
3,269,881	8/1966	Jones et al.	156—18 XR
3,293,093	12/1966	Alderuccio et al.	156—20 X

OTHER REFERENCES

Camp: A Study of the Etching Rate of Single-Crystal Germanium, in Journal of the Electro Chemical Society, vol. 102, No. 10, 1955, pp. 586-93.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,369,914

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Rodolphe Lacal

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

In the heading to the printed specification, line 4, "Dodolphe Lacal" should read -- Rodolphe Lacal --.

Signed and sealed this 12th day of August 1969.

(SEAL)

Attest:

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Commissioner of Patents